HS-TD G1888

Headspace-Thermal Desorption System

Operators Manual

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VERSION 1.2

MARKES international

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1. Introduction

Markes International's HS-TD system brings together two of the most powerful GC introduction techniques: equilibrium headspace (HS) and thermal desorption (TD). The combined system offers users optimum sensitivity for trace VOCs in solid, liquid and vapour-phase samples - all on one, versatile analytical platform.

This manual gives details of the Markes HS-TD system for the following models:

Agilent G1888 headspace system

Markes UNITY(e) thermal desorption system

2. Packing List

Remove the contents from the shipping box, inspect and check each item against the **Packing List** included with the order. Retain the instrument packaging and reuse if ever the system is to be shipped using conventional carriers.

3. Installing UNITY(e) onto G1888 headspace system

Take the aluminium tube (p/n SERUTD-1384) and insert into the PTFE collar (p/n SERUTD-1229). Also insert the three screws supplied (p/n SERZ-M416PPSS) into the PTFE block as shown in figure 1.

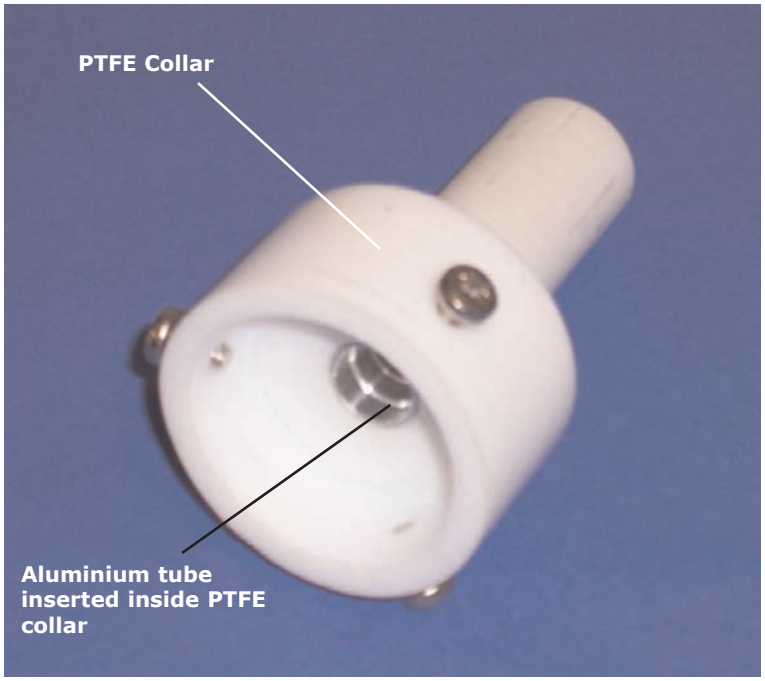

Figure 1: PTFE collar with aluminium tube inserted

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Take the UNITY-HS link tube assembly (p/n SERUTD-5132) (figure 2) and insert the end with the zero dead volume silcosteel union attached, into the PTFE collar as shown (figure 3).

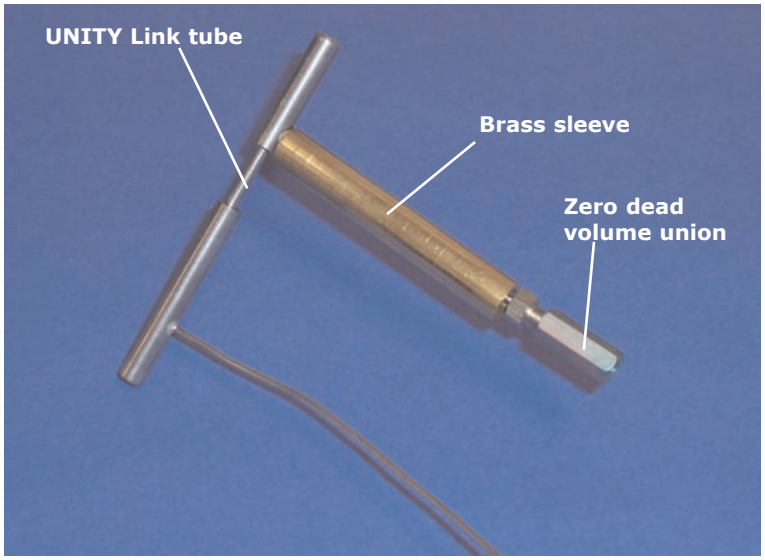

Figure 2: UNITY-HS link tube assembly

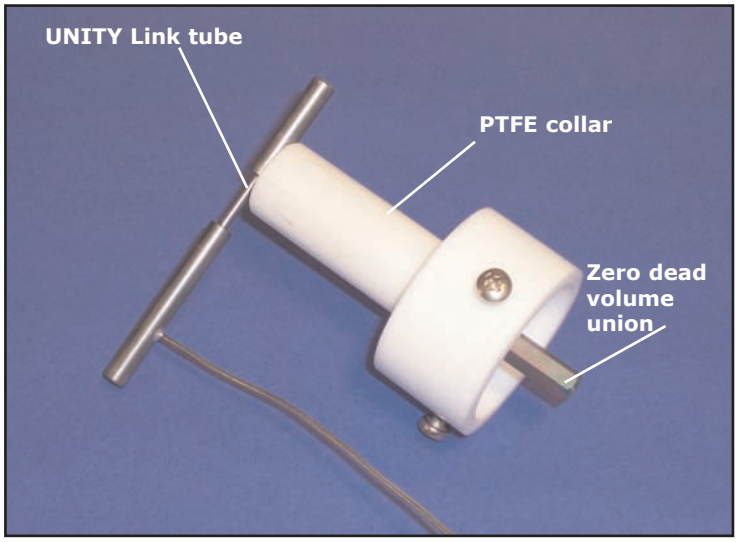

Figure 3: Link tube inserted into PTFE collar

Insert the link tube into UNITY (with the PTFE collar to the back of the system), but do not seal the link tube into place. Connect the HS transfer line into the union as shown (figure 4).

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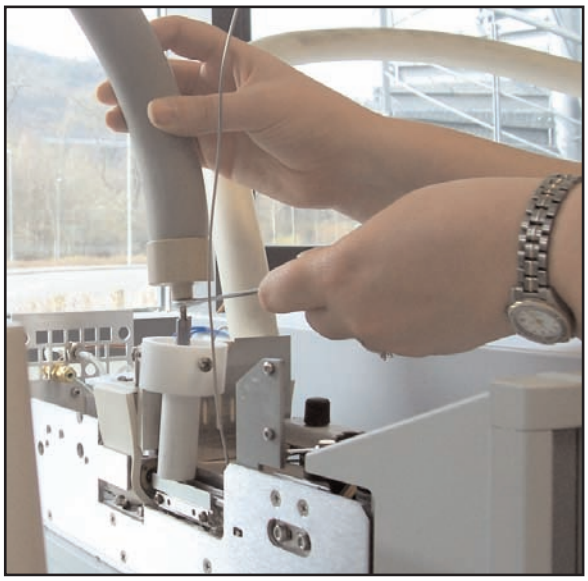

Figure 4: Connecting the HS transfer line to the inert zero dead volume union

Now secure the transfer line into the PTFE collar using the three screws as shown (figure 5).

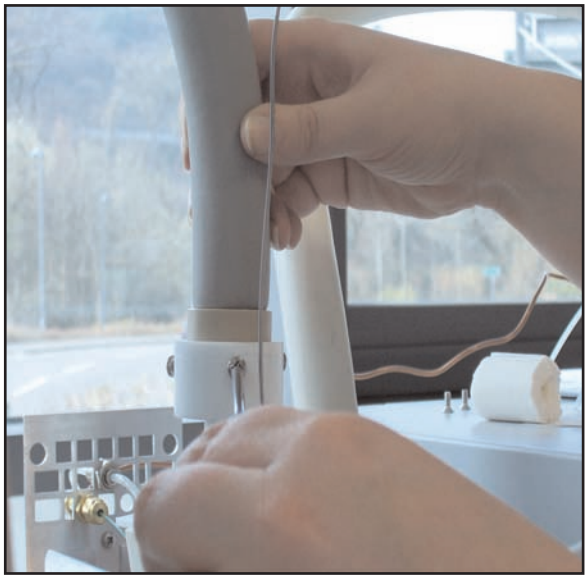

Figure 5: Secure the transfer line into the PTFE collar

Seal the UNITY link tube into the UNITY oven and fit the Perspex cover supplied (p/n SERMTD-1368) onto UNITY as shown (figure 6).

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Figure 6: Fit the perspex cover supplied

Connect the 1/16 inch stainless steel union supplied (p/n SERZ-0386) to the carrier gas inlet tube on the UNITY link tube. Then connect the 500 mm piece of 1/16 inch stainless steel tubing supplied (p/n SERZ-0175) to the union in order to extend this inlet gas line (figure 7).

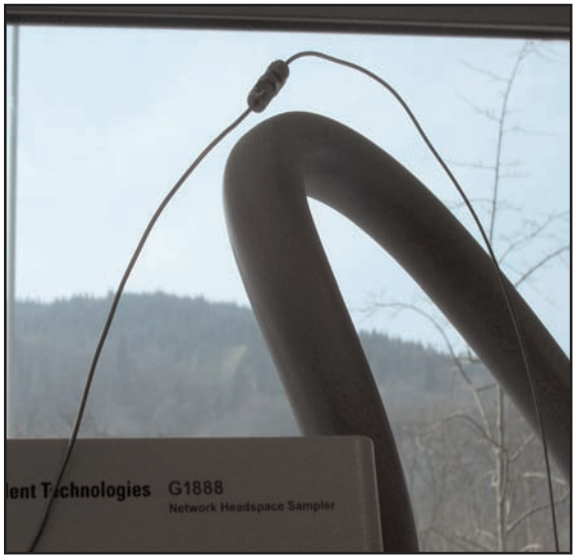

Figure 7: Extending the carrier gas inlet line

Open the top cover of the HS unit, and remove the stainless steel cover over the valve system by loosening the two screws and lifting off the cover (figure 8).

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Figure 8: Removing the HS valve cover

Unscrew the screw and remove the black cover over the headspace needle, to expose the silcosteel union (figure 9).

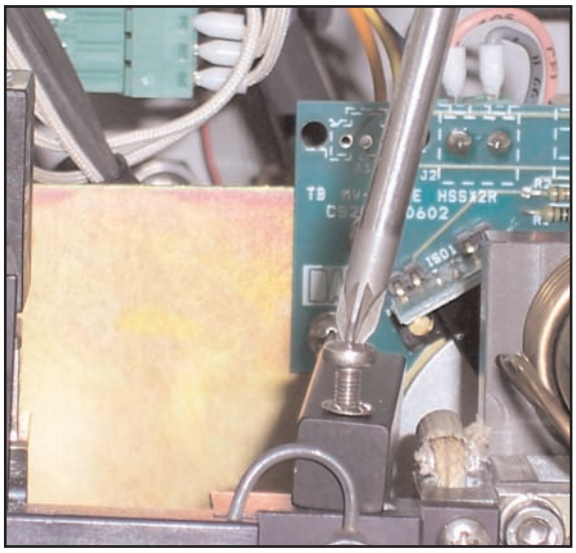

Figure 9: Removing the black needle cover

Remove the silcosteel tubing from the union as shown (figure 10).

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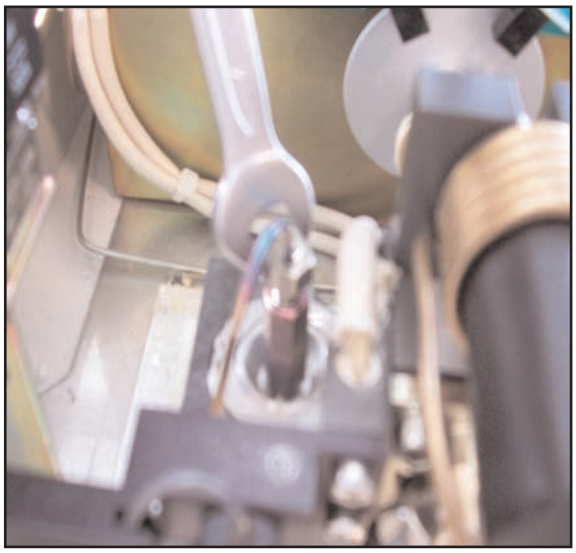

Figure 10: Removing the silcosteel tubing

Also remove the silcosteel tubing from the 6-port headspace valve and retain for use if you wish to return to standard headspace operation in the future.

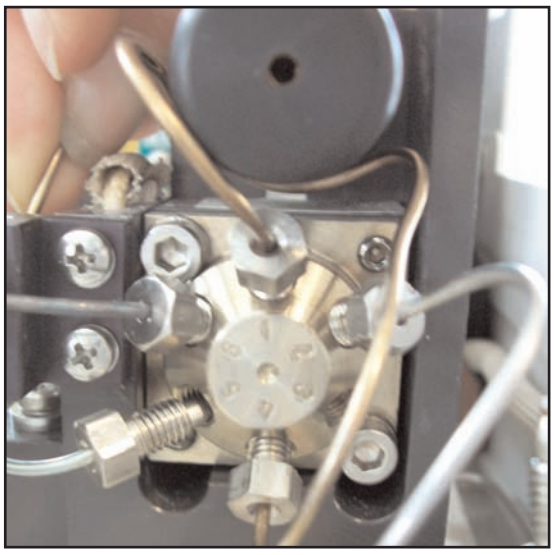

Figure 11: Removing the silcosteel tubing from the 6 port valve

Connect the 170 mm length of 1/16 inch silcosteel tubing supplied (p/n SERZ-0679) to the headspace needle and feed through as shown in figure 12.

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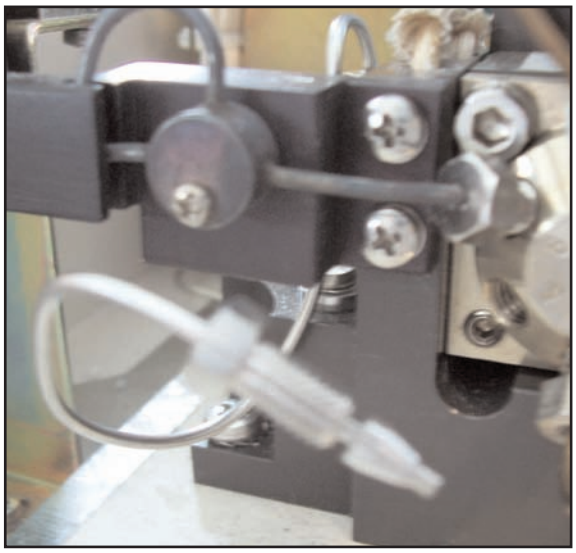

Figure 12: Connecting 1/16 inch silcosteel tubing

Remove the HS fitting which connects the 6-port valve to the HS transfer line and position the sample outlet line in a vertical position as shown (figure 13).

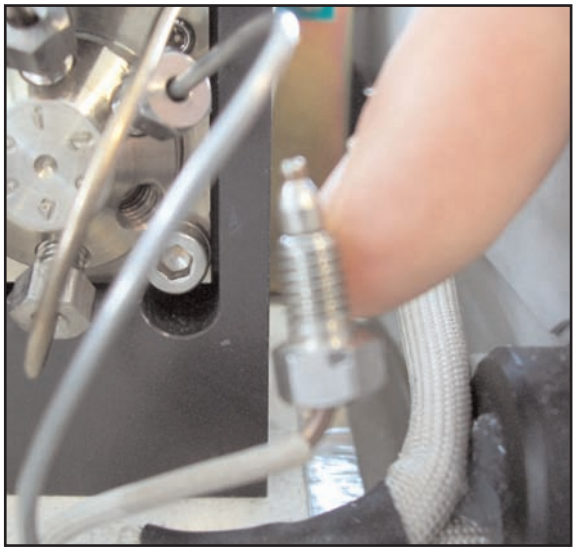

Figure 13: Position sample outlet line vertically

Connect 100 mm length of 1/16 inch silcosteel tubing supplied (p/n SERZ-0679) to the zero dead volume connector (p/n SERZ-0691) and connect this union to the sample outlet line as shown (figure 14).

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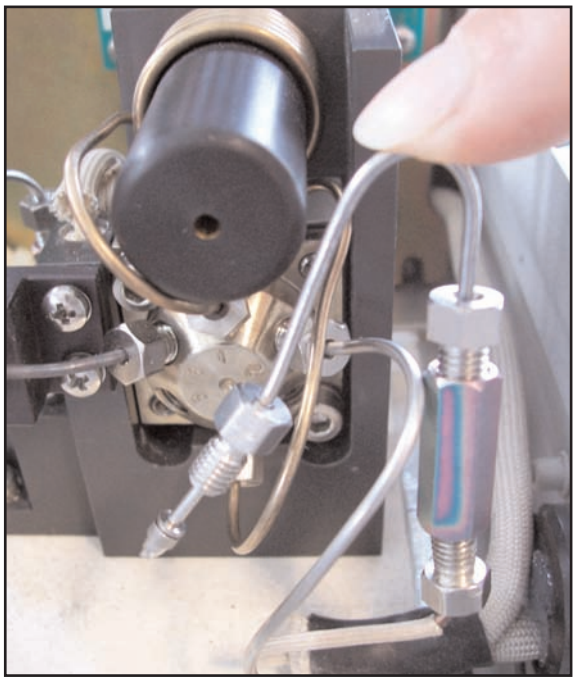

Figure 14: Connecting the sample outlet line

Connect both the lengths of 1/16 inch silcosteel tubing connected above, into the silcosteel zero dead volume Valco T-piece (p/n SERZ-0699) as shown in figure 15.

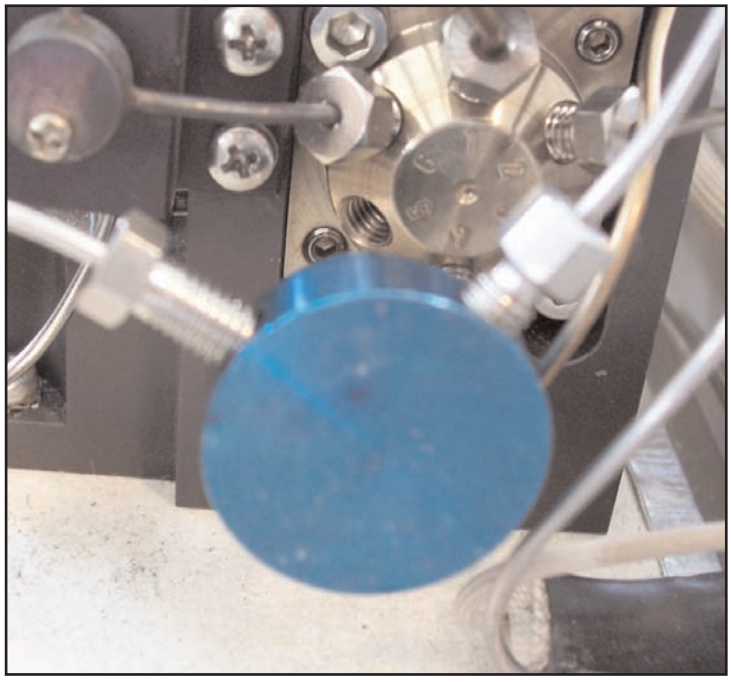

Figure 15: Connecting up the Valco T-piece

Connect the 1/16 inch stainless steel tubing from the UNITY link tube into the Silcosteel T-piece as shown (figure 16).

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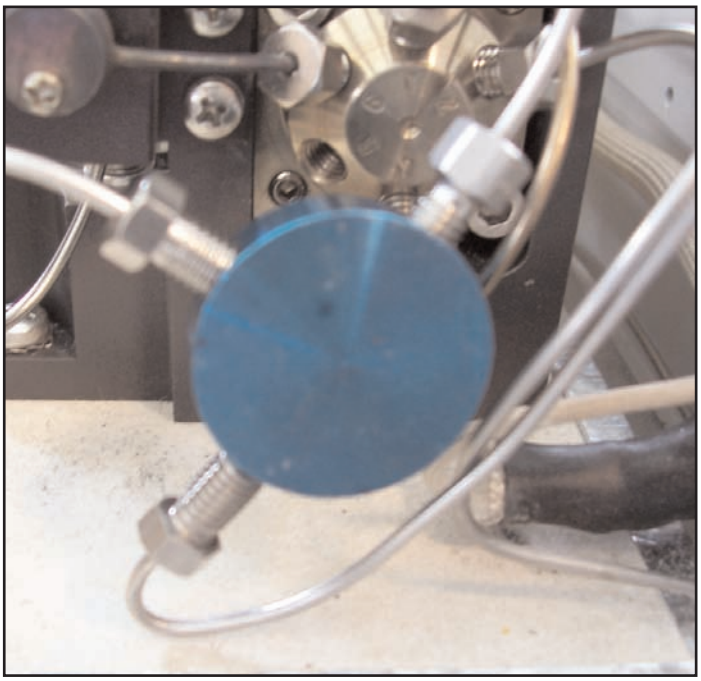

Figure 16: Connect the UNITY line into the T-piece

Replace the black needle cover over the Headspace needle connection and the metal cover over the headspace valve system.

Finally feed the carrier gas inlet line to the front right hand corner of the Headspace unit and carefully shut the headspace cover taking care to ensure that the cover does not crimp the gas line (figure 19).

Figure 17: Gas line protruting from HS system

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This completes all the flow path connections.

To leak test the system, place an empty headspace vial into position 1 in the G1888 and set to "Inject" with a long sampling time (sufficient to allow full leak testing).

Use the UNITY "Leak test" dialogue, to pressurise the system and test for leaks.

4. Software Instructions

Prior to installing software, the Headspace system should be connected *via* a LAN cable to the network, and a spare IP address should be available.

Using the HSS menu function:

Check LAN settings

Sub net mask must be the same as on the operating PC. This can be checked on the PC by doing the following:

Click on 'Run' from the Start menu
Type CMD in the box
When command prompt is launched, type in "ipconfig /all"
The subnet mask will be displayed.

Installing s/w

Load CD and copy folder to Program directory on PC.
Open executable file Agilent G1888.exe

This will open the HSS operating software.

Connecting to HS

Select TCP/IP from the "remote" menu.
This will open a TCP/IP Connection window.
Insert the IP address that you will be running the HSS from.
Click connect.
The TCP status should change from yellow to green, and state online.

Setting up UNITY for operation with G1888 HS system

Select the "Unity.opt" file, in the UNITY program folder.
Open this file using notepad.
In this text file, change the following lines:
Direct = off should be changed to ***Direct = on***
Syncstatdisplay = Probe in should be changed to ***Syncstatdisplay = start sampling***
Save the changes to this file, close, and restart UNITY.

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From the View>Options menu, select the configuration tab.
Tick the box which says operate direct mode with HP7694.
Close software.

Open HS operating software and connect to the unit as above.
Open UNITY s/w
When you select Direct sampling from the drop down menu, a new window should appear which says HP7694DDE.

This completes the software installation and setup.

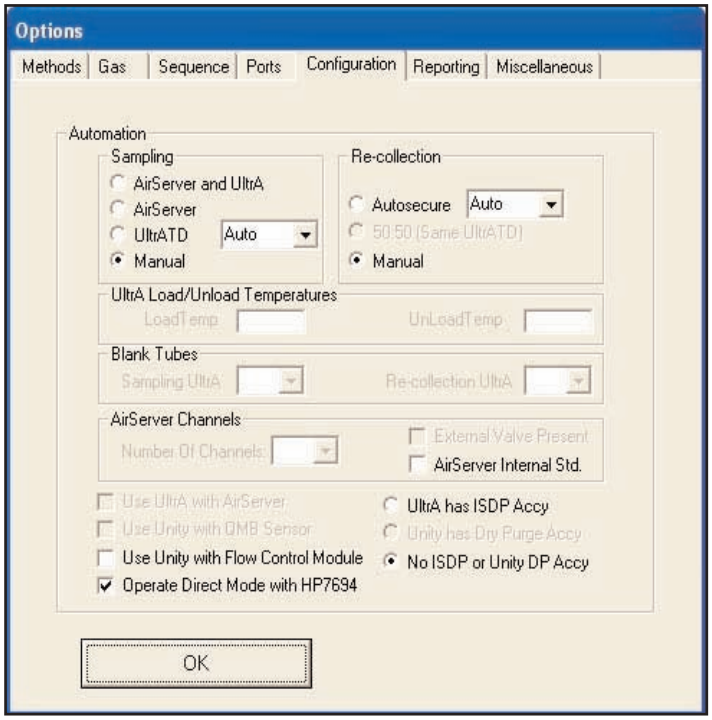

Figure 18: UNITY "Options" menu, displaying selection to operate direct mode with HP 7694

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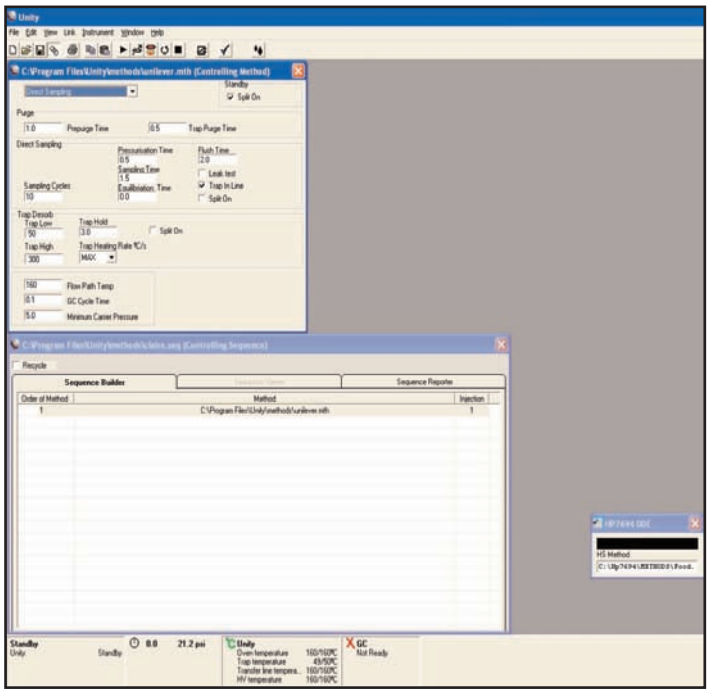

Figure 19: UNITY operating software displaying status of HS system

5. Operation

5.1. Headspace settings

The HS parameters are controlled *via* the "Method Parameter" window in the G1888 controlling software (see figure 20).

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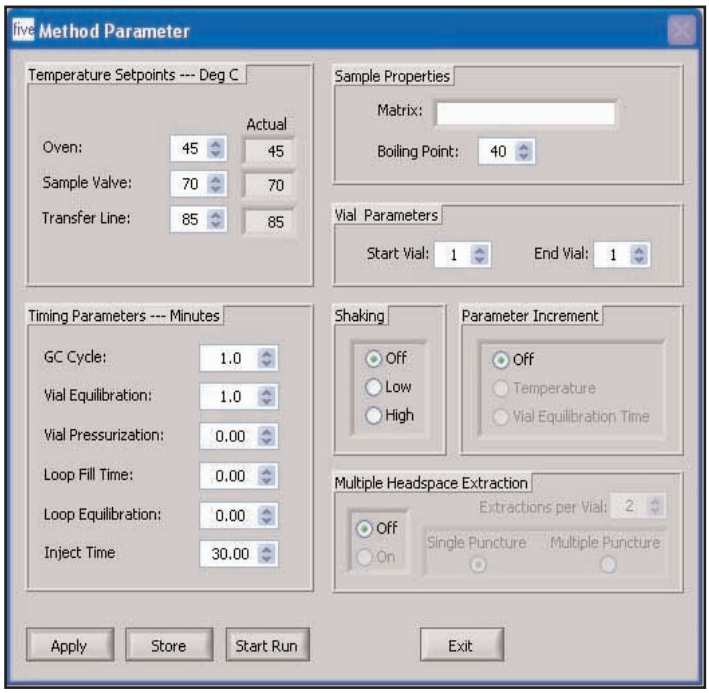

Figure 20: Method parameter window for controlling HS sampler

Oven Controls temperature of HS vial oven
Sample Valve Controls temperature of sample valve
Transfer line Controls temperature of HS transfer line

GC Cycle Sets interval between runs to allow overlap of samples. For HS-TD operation, the total GC cycle time should be calculated as follows:

GC runtime + GC equilibration (cooldown) time + HS injection time

Vial equilibration: Sets time for equilibration of vial in HS oven
Vial pressurization: Not used in HS-TD operation
Loop fill time: Not used in HS-TD operation
Loop equilibration: Not used in HS-TD operation

Injection time: Sets the time for which the HS needle is inserted into the vial to allow sampling. This is calculated from the UNITY method conditions, and is the total of all purge times and direct sampling times: (Note: if multiple extractions are being carried out, it is important to calculate the correct total time for direct sampling)

Vial Parameters: Sets the sequence of vials to be sampled

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5.2. UNITY settings

Note: Dynamic direct sampling involves a continuous flow of carrier gas through (or from) the sample vessel sweeping the headspace vapours into the UNITY cold trap for a defined period of time and is not compatible with single needle headspace systems.

Pulsed direct sampling works by pressurising a sealed sample vessel for a set period of time and then shutting off the gas supply and allowing the carrier gas pressure to be released out through the cold trap of UNITY. This process may be repeated multiple times on a single sample to enhance the concentration of trapped analytes before desorbing the cold trap and beginning the GC(/MS) analysis. Pulsed direct sampling is thus compatible with a single inlet/outlet point and can therefore be used with single needle headspace systems

Sampling parameters

Prepurge:

Prepurge pressurises the sample vessel and cleans out the sampling lines thus helping prevent carryover from previous runs and sweeps any air out of the flow path.
However, as there is no overall flow in the sampling vessel prepurge does not flush the vessel. Prepurge therefore defines the time for which gas sweeps through the sample lines and into UNITY
through the flow-path bypassing the trap and out through the split at the flow rate set on the split needle valve

Pressurisation time:

When set to zero UNITY runs in dynamic direct sampling mode.

When set to any non-zero value UNITY will run in pulsed rather than dynamic direct sampling mode.
When in pulsed sampling mode this defines the time for which carrier gas is supplied to the sample vessel to build up pressure prior to releasing that pressure through the cold trap.

Sampling time:

Defines the amount of time for which the solenoid valve downstream of the cold trap is opened so that sample is transferred from the sample vessel to the cold trap. The pressure drops as soon as sampling begins until it eventually reaches atmospheric pressure.

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Equilibration time:

Governs the amount of time for which the sample in the sampling vial is allowed to re-equilibrate before it is repressurised and resampled.

Sampling cycles:

Sets the number of times that the pressurisation, sampling and equilibration steps will be repeated for any particular sample, before desorption of the focusing trap triggers the GC(/MS) analysis.

Flush time:

A similar step to Prepurge, this stage permits carrier gas purge of the sample lines to remove any residual sample and transfer to the trap or out of the split or both.

Trap Purge:

Defines the time for which carrier gas is purged through the cold-trap in the sampling direction. This procedure purges the trap of any oxygen prior to heating and, under certain conditions, allows selective purge of unwanted volatiles such as water or ethanol.

GC Cycle

The GC cycle time should be set to 0.1 for all HS-TD operations.

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